Silicon-Based Resonant Microsensors

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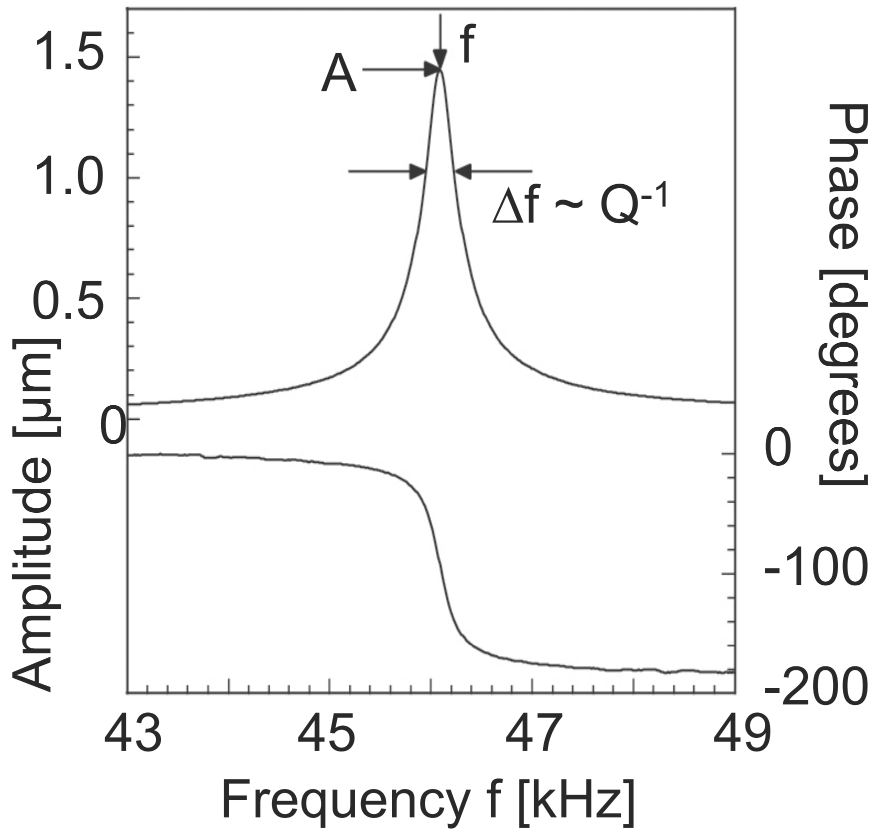
Outline

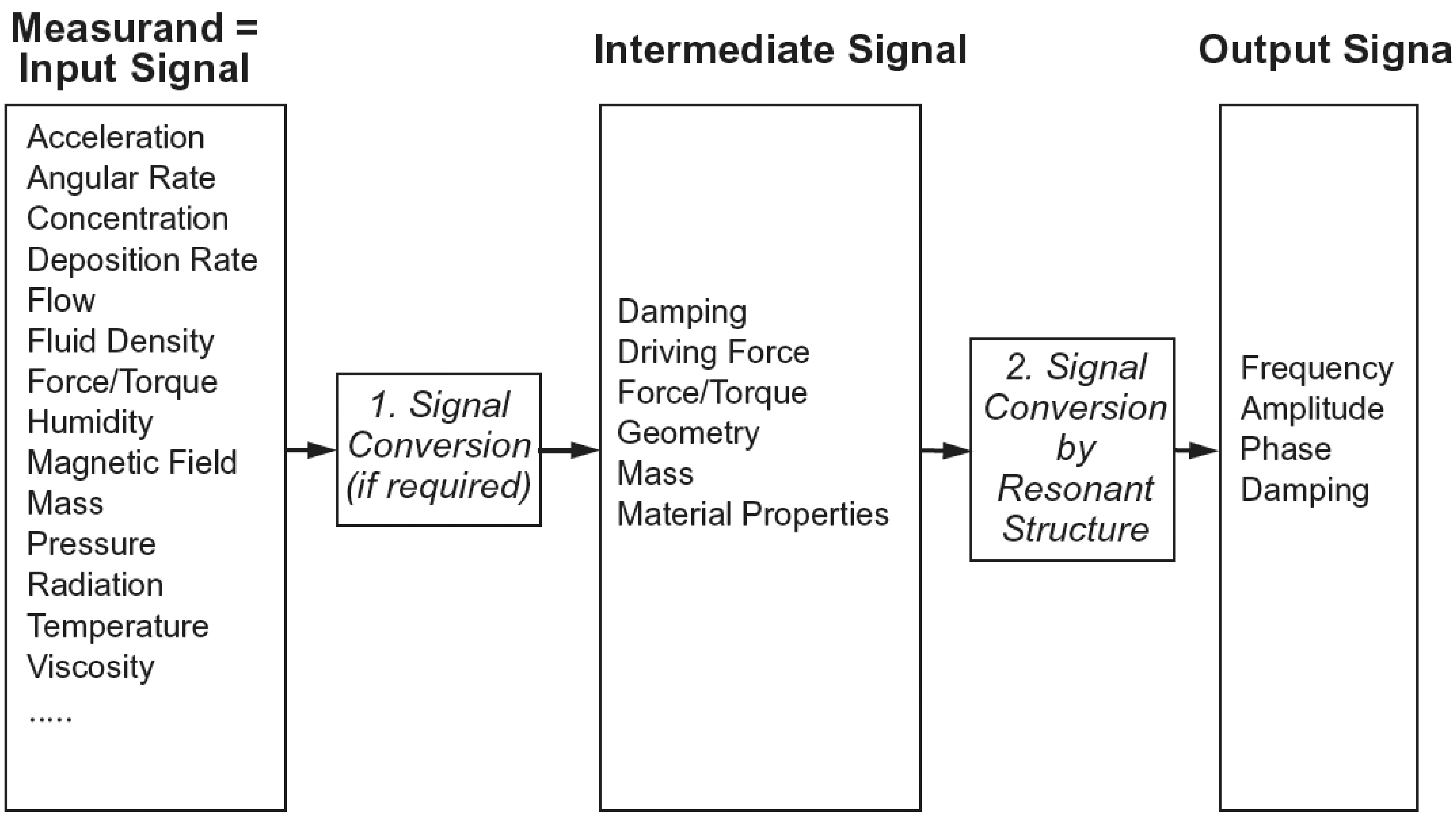
- Resonant Microsensors
  - Operation Principles and Applications
- Frequency-Output Sensors
  - Operation in Feedback Loop
  - “Device” vs. “System-Level” Resonant Sensor
  - Frequency Resolution and Drift
  - Drift Compensation Techniques
- (Bio)Chemical Resonant Microsensor Platform
  - Disk-Type Microstructure Optimization
  - Chemical Sensing
- Summary and Outlook

Resonant Sensors

- Measurand affects characteristic of resonant behavior of microstructure
  - **Resonance frequency**
  - Quality factor
  - Vibration amplitude
  - Phase

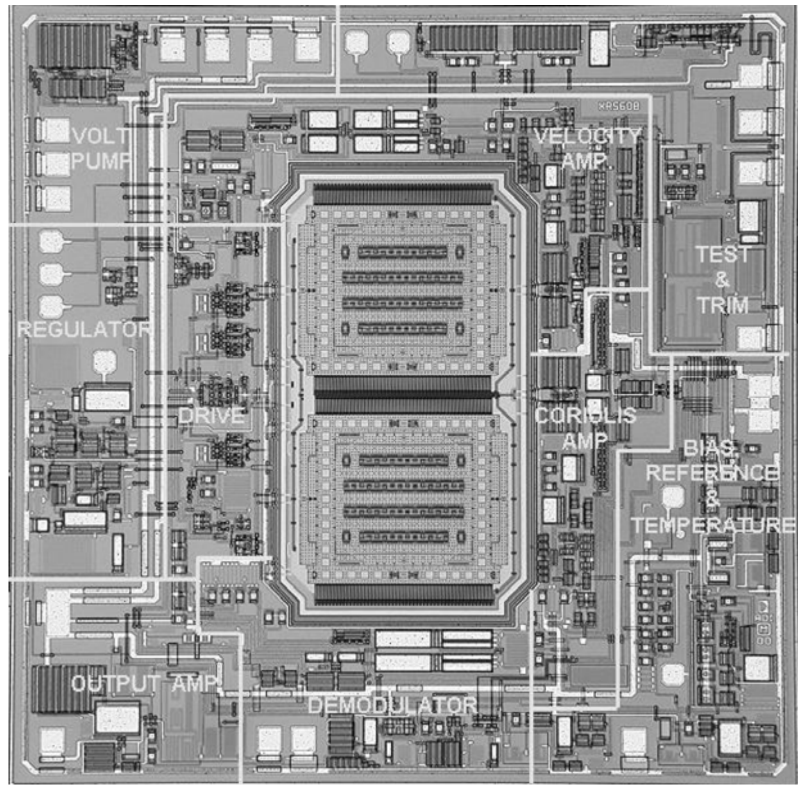


Resonant Sensors



Why Resonant Sensors?

- Resonant Sensors
  - Quasi-digital output signal
  - Frequencies can be measured precisely
- If Combined with CMOS Technology
  - Established fabrication base
  - On-chip circuitry
  - Fabrication outsourcing



Analog Devices ADXRS 150

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Frequency-Output Sensors
– Working Principle –

$$m\frac{d^2x}{dt^2}+b\frac{dx}{dt}+k\,x=\sum F_i$$

- **Device-Level** Frequency Sensor

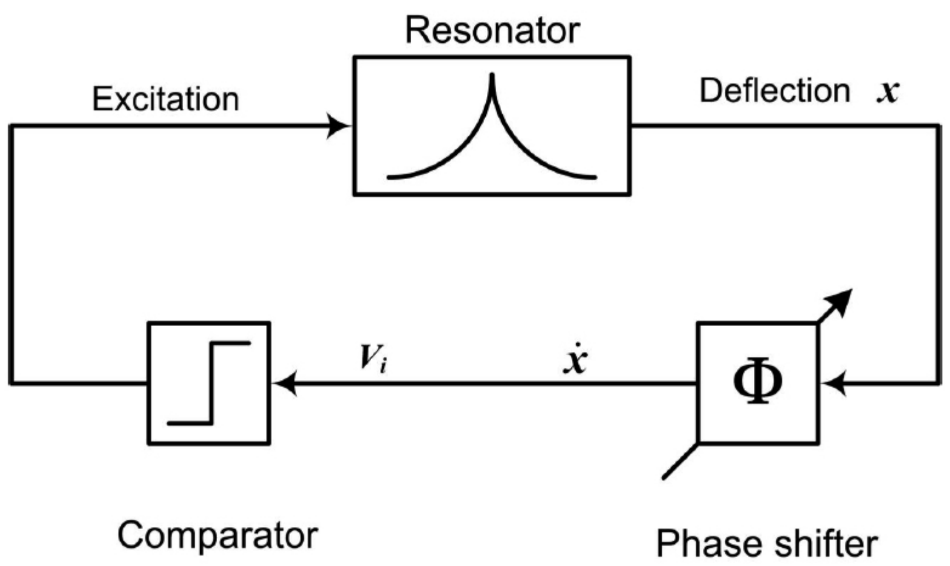
$$F=F_1=b\frac{dx}{dt} \Rightarrow f=\frac{1}{2\pi}\sqrt{\frac{k}{m}} \Rightarrow \frac{\Delta f}{f}=\frac{1}{2}\left[\frac{\Delta k}{k}-\frac{\Delta m}{m}\right]$$

- **System-Level** Frequency Sensor

$$F=F_1+F_2+F_3=b\frac{dx}{dt}+\boxed{k_{add}\,x+m_{add}\frac{d^2x}{dt^2}} \Rightarrow f=\frac{1}{2\pi}\sqrt{\frac{k-k_{add}}{m-m_{add}}}$$

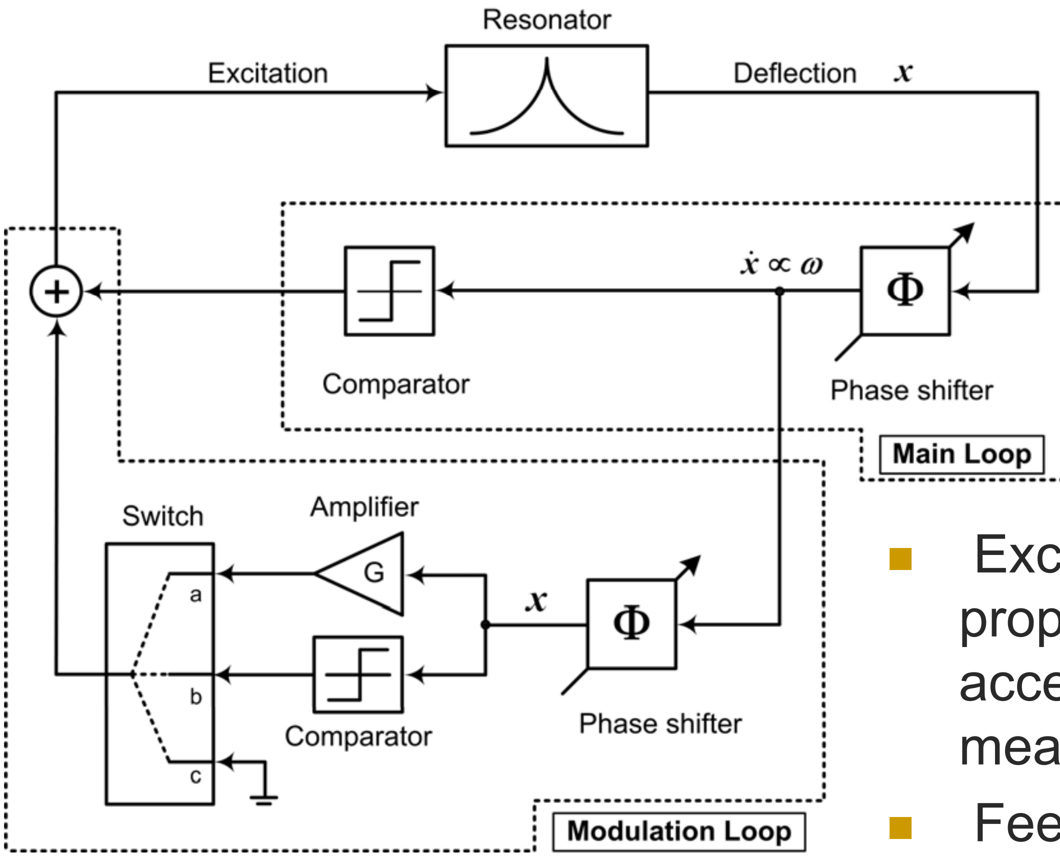
$$\frac{\Delta f}{f}=\frac{1}{2}\left[\frac{m_{add}}{m}-\frac{k_{add}}{k}\right]$$

Device-Level Frequency Sensor



- In steady state, the excitation force compensates for damping loss and microstructure vibrates with constant amplitude
- Changes of the effective spring constant or mass result in a measurable frequency change

System-Level Frequency Sensor



- Excitation force component proportional to deflection (or acceleration) is modulated by measurand
- Feedback loop introduces measurable frequency change

Resonant Magnetic Field Sensor – Working Principle –

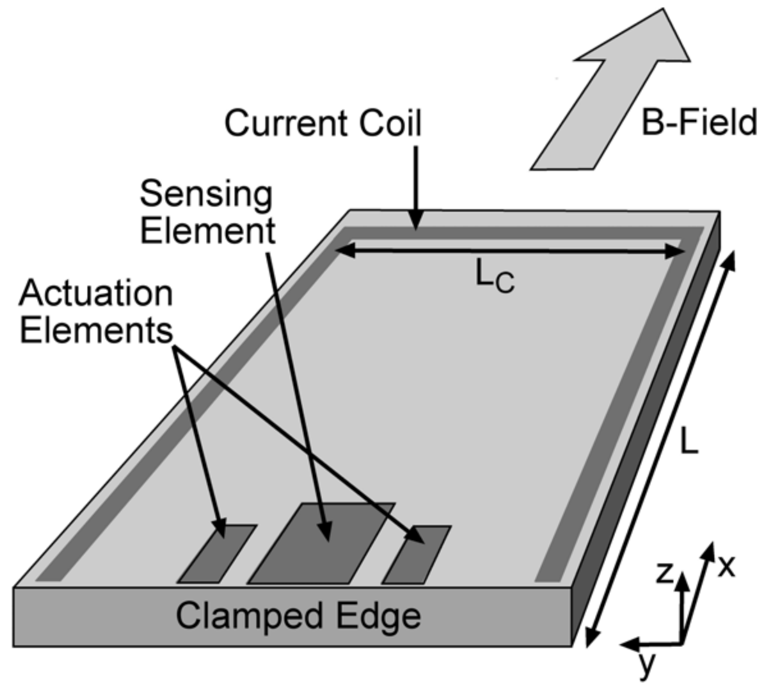
- Current through coil $I = k_B\, x$ generates Lorentz force F_L proportional to the cantilever deflection x in presence of magnetic field

$$F_L = q\left(v \times B\right) = I\, L_C B = k_B x\, L_C B$$

- Assume: resonator damping compensated by thermal bimorph actuation

$$m\frac{d^2x}{dt^2} = -kx + F_L = (k_B\, L_C B - k)x$$

$$\Rightarrow \quad \boxed{f = \frac{1}{2\pi}\sqrt{\frac{k}{m}} = \frac{1}{2\pi}\sqrt{\frac{k - k_B L_C B}{m}}}$$



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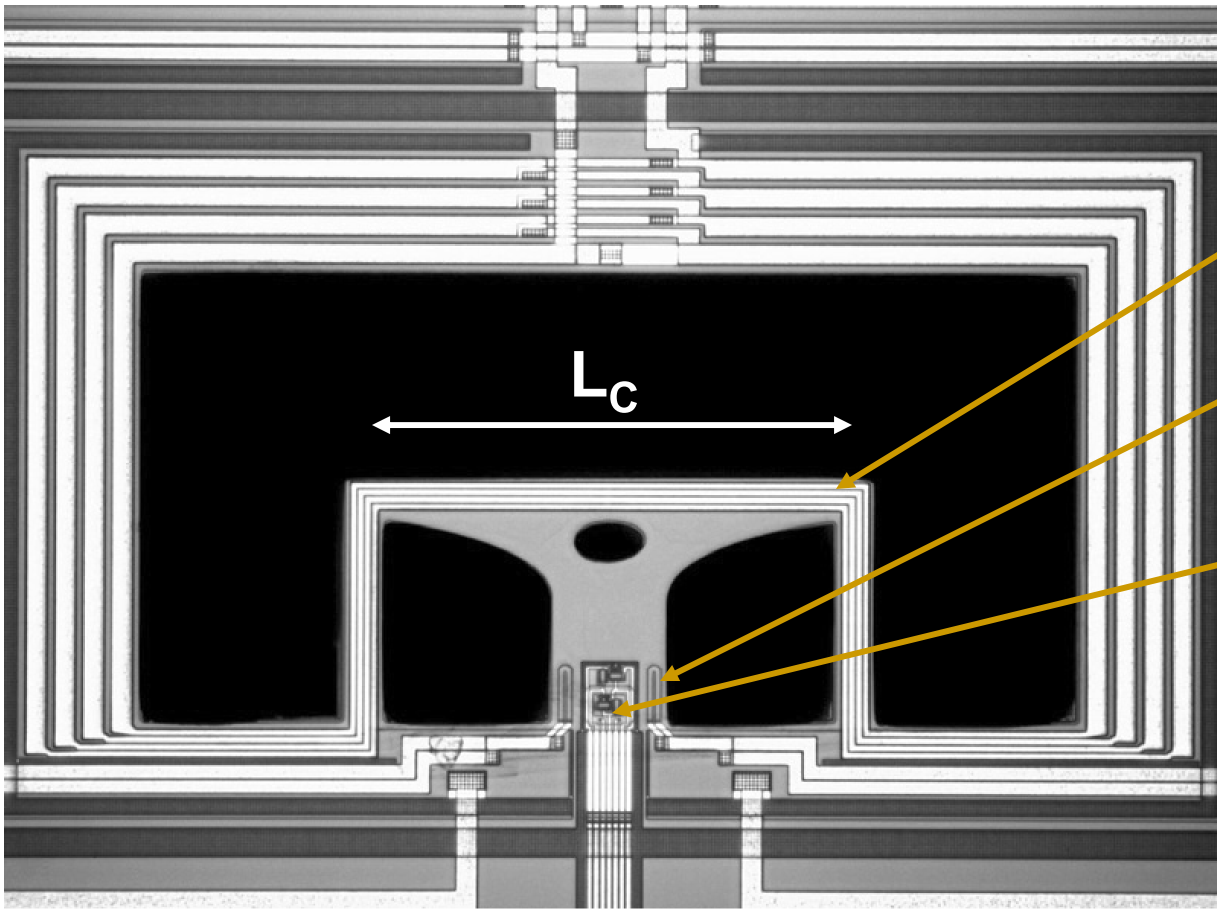
Resonant Magnetic Field Sensor
– Microstructure Design –



R. Sunier et al.,
IEEE J. MEMS,
15 (2006) 1098-1107

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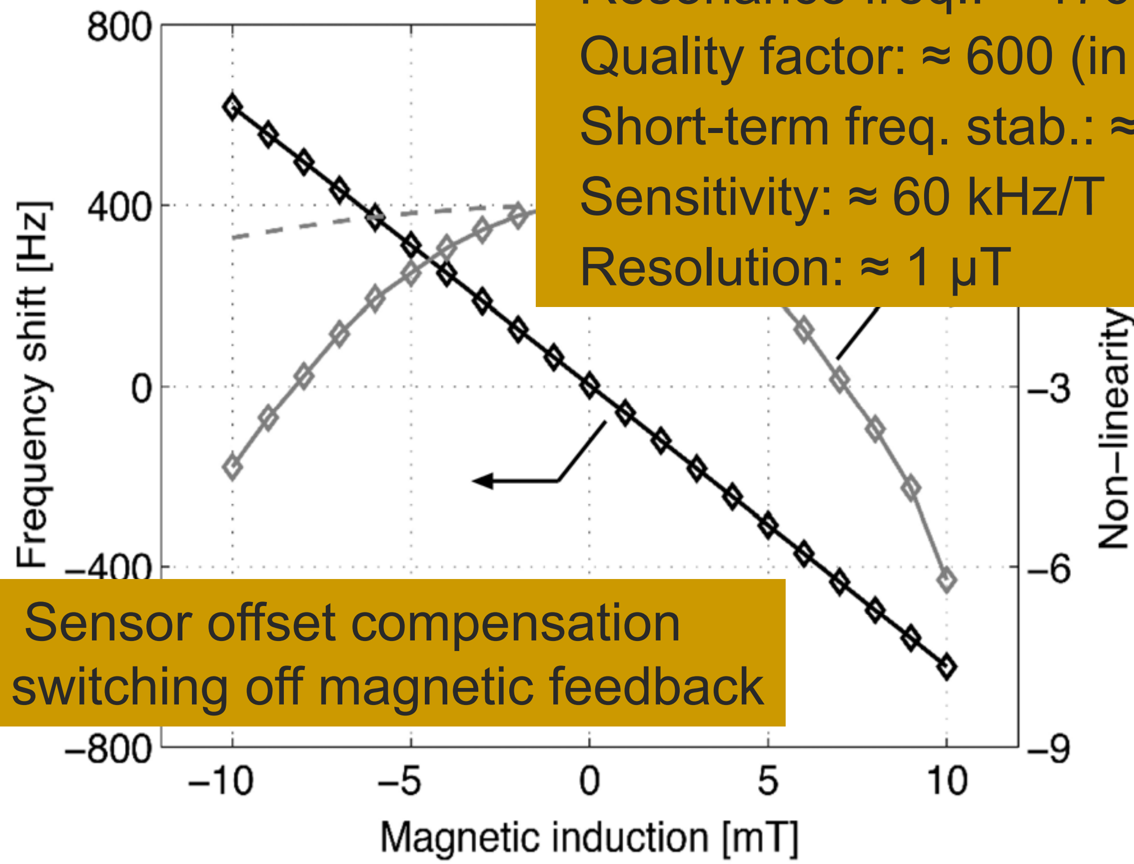
Magnetic Field Detection – Sensor Sensitivity –

Resonance freq.: ≈ 176 kHz
Quality factor: ≈ 600 (in air)
Short-term freq. stab.: ≈ 25 mHz
Sensitivity: ≈ 60 kHz/T
Resolution: ≈ 1 μT



Sensor offset compensation
by switching off magnetic feedback

Resonant Sensor Resolution

- **Sensor resolution** is limited by sensor sensitivity S [Hz/"measurand change"] and minimal detectable frequency change Δf_{min} [Hz]

$$c_{min} = \frac{\Delta f_{min}}{S}$$

- **Limit of Detection (LOD)** is measurand amplitude that generates three times the noise amplitude, i.e.

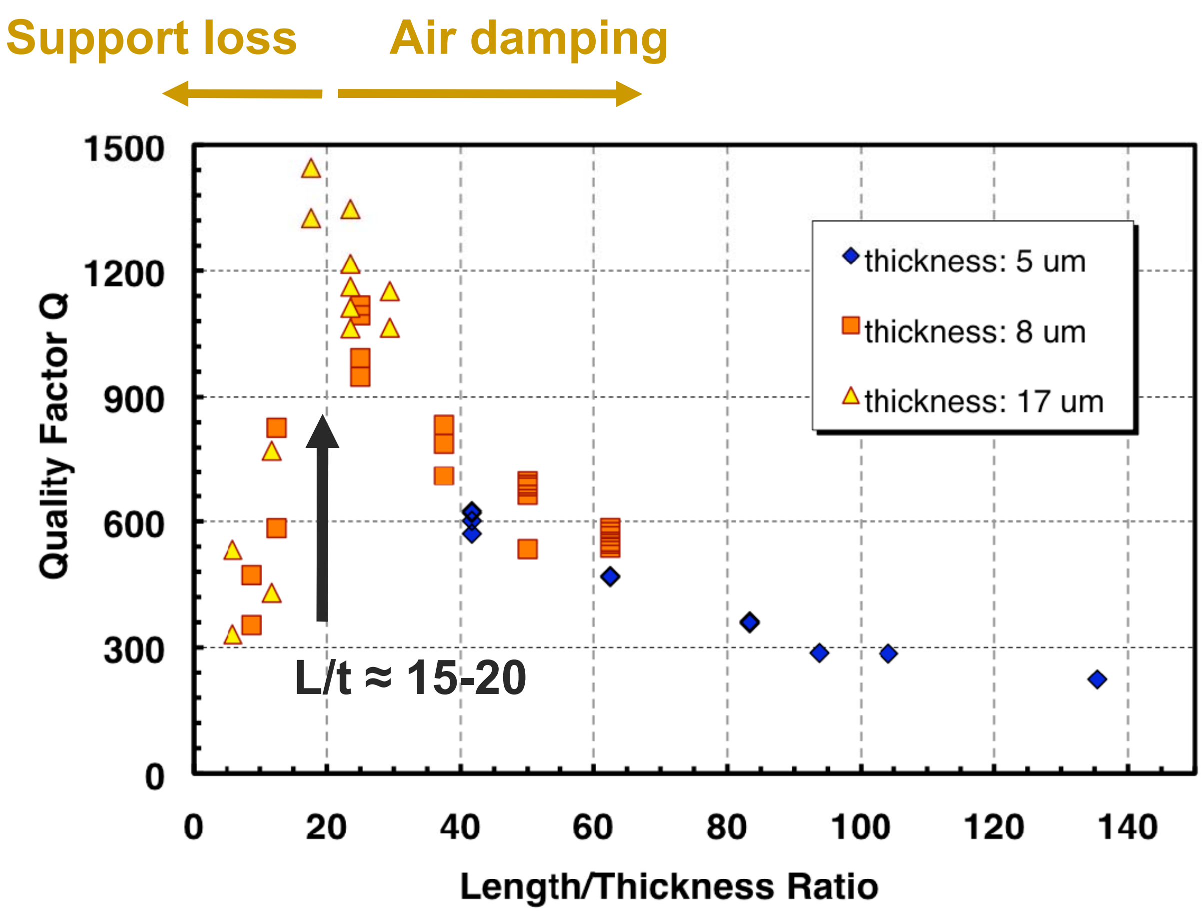
$$LOD = 3\,\frac{\Delta f_{min}}{S}$$

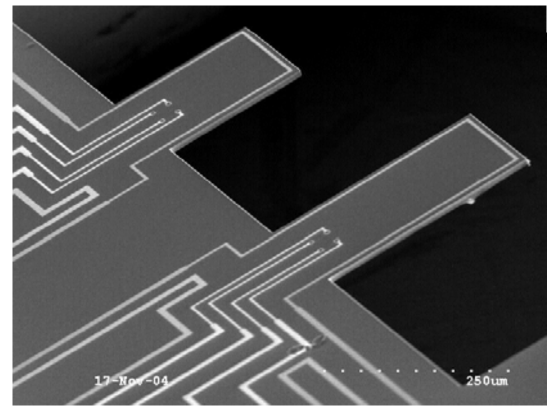
- Minimizing Δf_{min} generally demands maximizing quality factor Q; e.g. for cantilever in amplifying feedback loop

$$\Delta f_{min} = \sqrt{\frac{f_0\, k_B\, T B}{2\pi\, k\, Q \left\langle x^2 \right\rangle}}$$

Albrecht et al., *J. Appl. Phys.* **69** (1991) 668

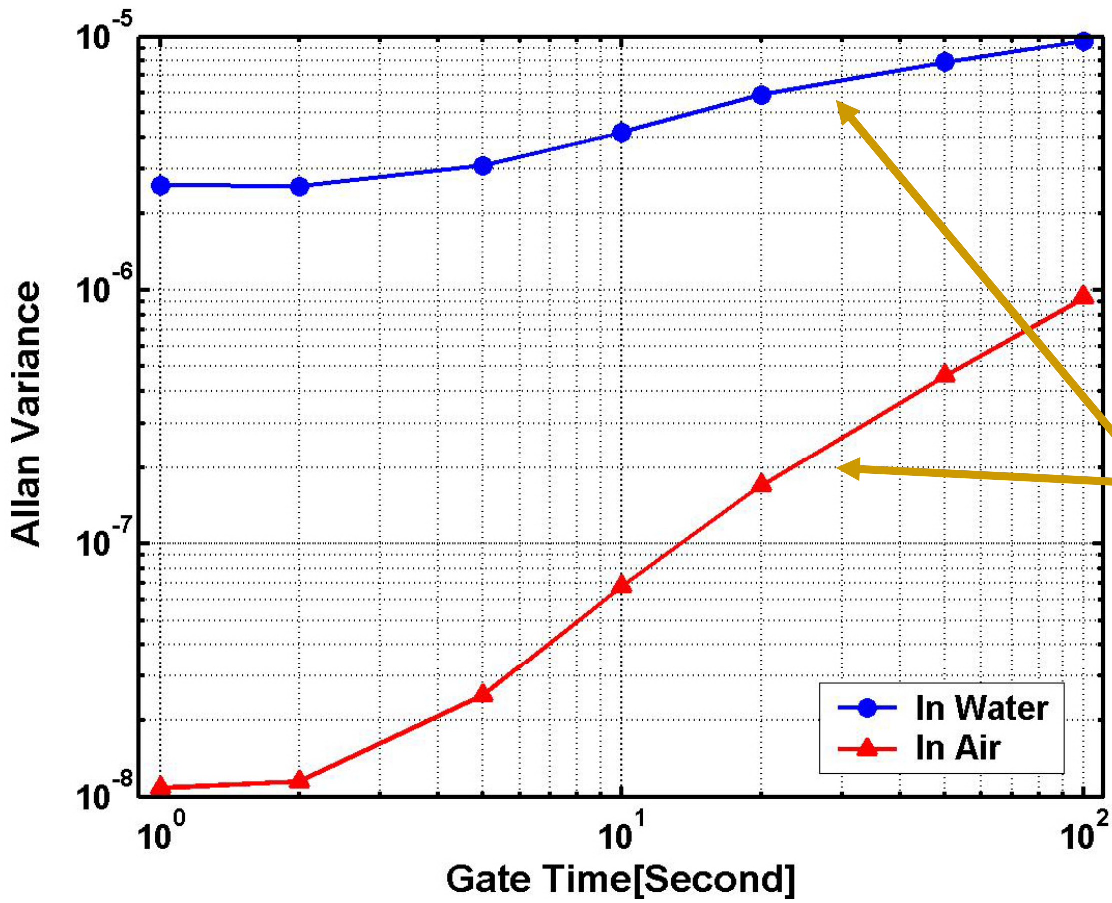
Silicon Cantilever Beam
– Q-Factor Optimization –

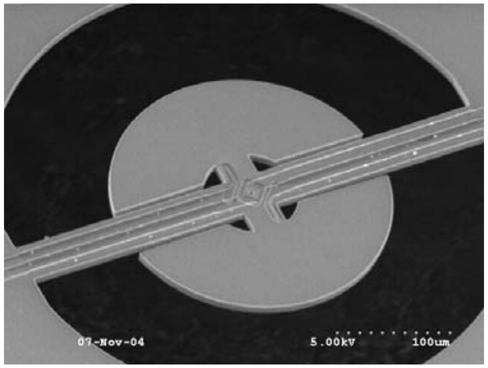




K. Naeli et al.,
Proc. Transducers '07, pp. 245-248

Silicon Disk-Resonators
– Allan Variance vs. Gate Time –





Increase due to Long-Term Drift

$$\sigma^2(\tau,m) = \frac{1}{2m}\sum_{n=1}^{m}(\gamma_{n+1} - \gamma_n)^2$$

$$\gamma_n = \frac{f_{n+1} - f_n}{f_n}$$

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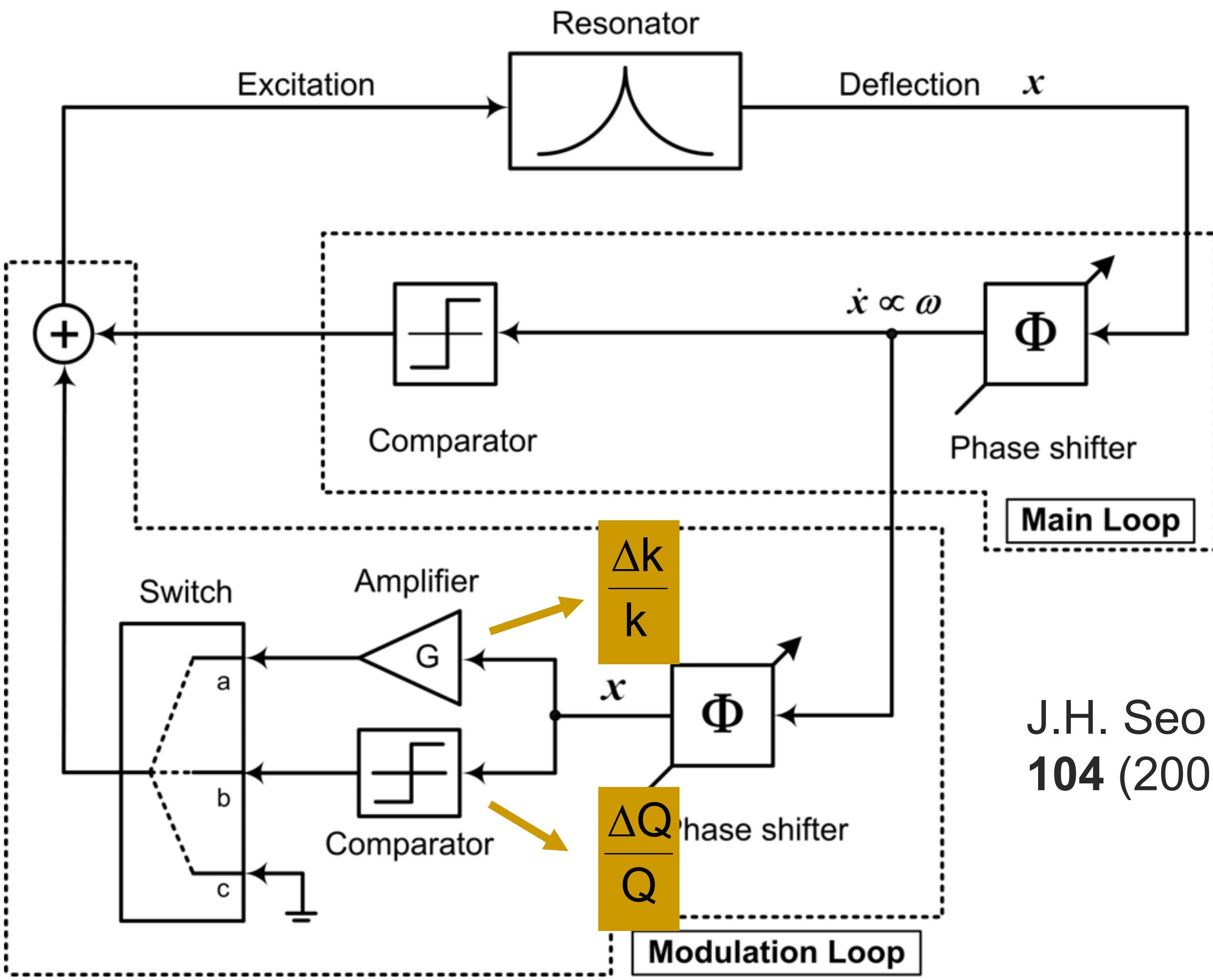
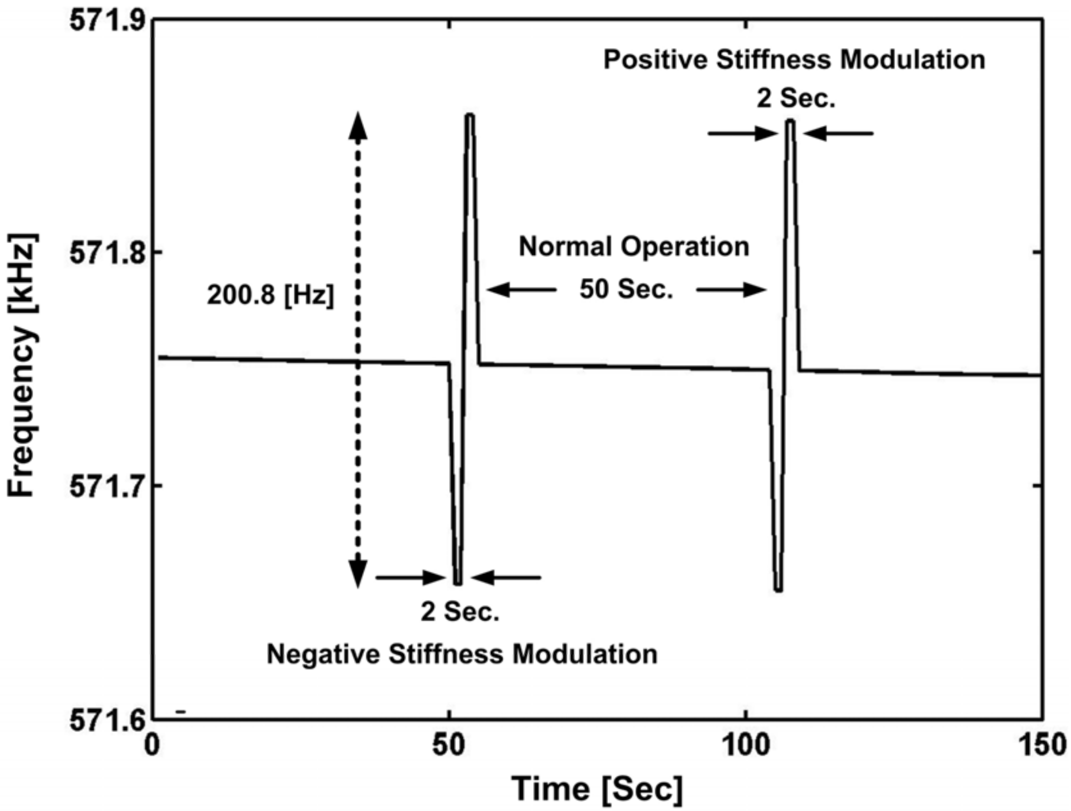
Drift Compensation: Method I
– Controlled Stiffness Modulation –

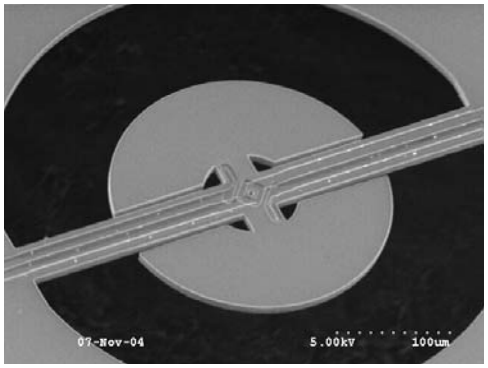


Compensation in closed-loop operation!

J.H. Seo et al., *J. Appl. Phys.* **104** (2008) 014911

Q-Factor Tracking via Periodic Stiffness Modulation





$$\left(\frac{\omega_{POS}}{\omega_{NEG}}\right)^2 = \alpha$$

$$Q = \frac{\alpha + 1}{\alpha - 1}$$

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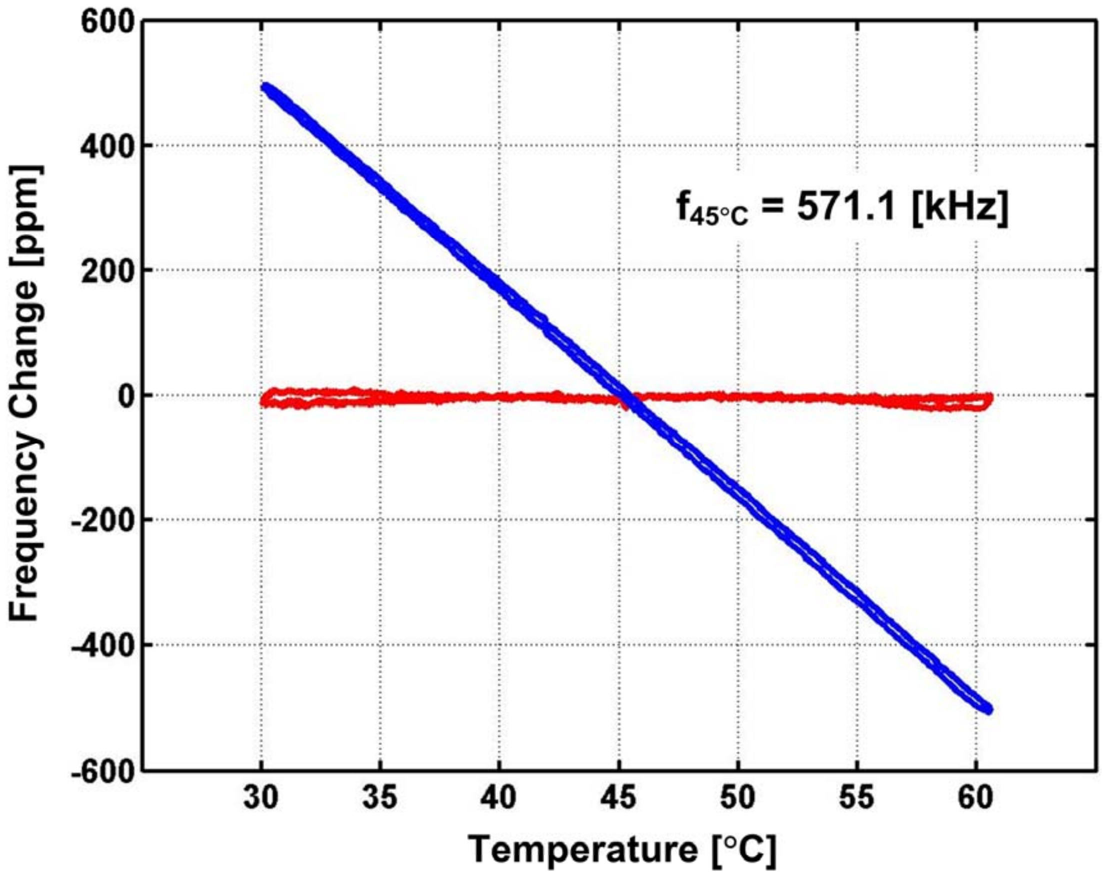
Temperature Compensation via Q-factor Tracking



Uncompensated:

df/dT ≈ - 33 ppm/˚C

Compensated:

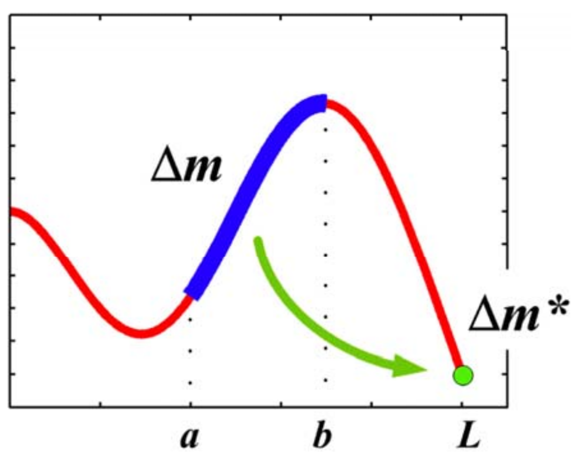
df/dT < 2 ppm/˚C

J.H. Seo et al,
Proc. Hilton Head Workshop,
pp. 190-193, 2008

Drift Compensation: Method II
– Overtone Analysis in Prismatic Beams –

- Goal: Compensation for environmental effects in cantilever-based mass-sensitive sensors
- Idea: Explore flexural resonances of **partly-covered** beams
- Key: Flexural resonance frequencies of prismatic, homogeneous beam only differ by λ_i

$$f_i = \frac{\lambda_i^2}{2\pi\sqrt{12}} \frac{t}{L^2} \sqrt{\frac{E}{\rho}}$$



- Assumption: added mass does not affect k!
- **Difference of relative frequency changes $\Delta f_i/f_i - \Delta f_j/f_j$ only depends on added mass but NOT on environmental changes!**

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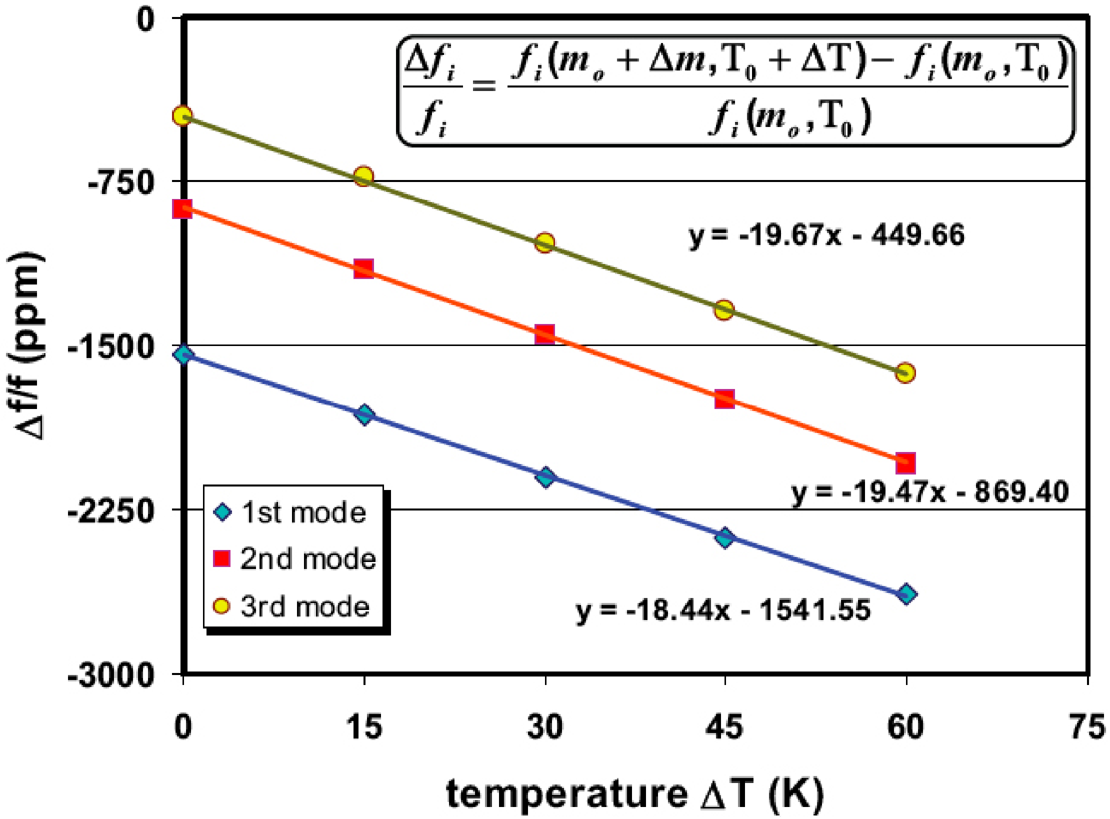
Temperature Compensation via Overtone Analysis



O. Brand, K. Naeli et al., *Proc. MME 2008 Workshop,* pp. 121-127, 2008

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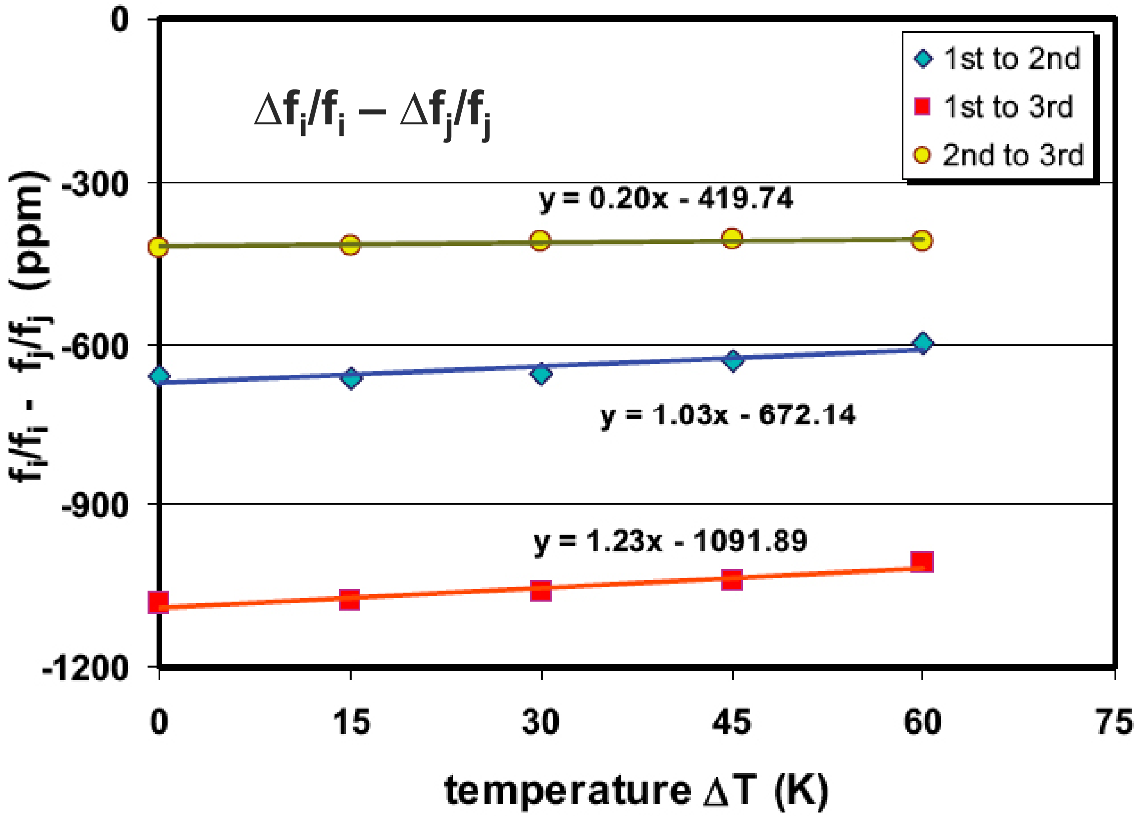
Temperature Compensation via Overtone Analysis



Uncompensated:

df/dT ≈ - 20 ppm/˚C

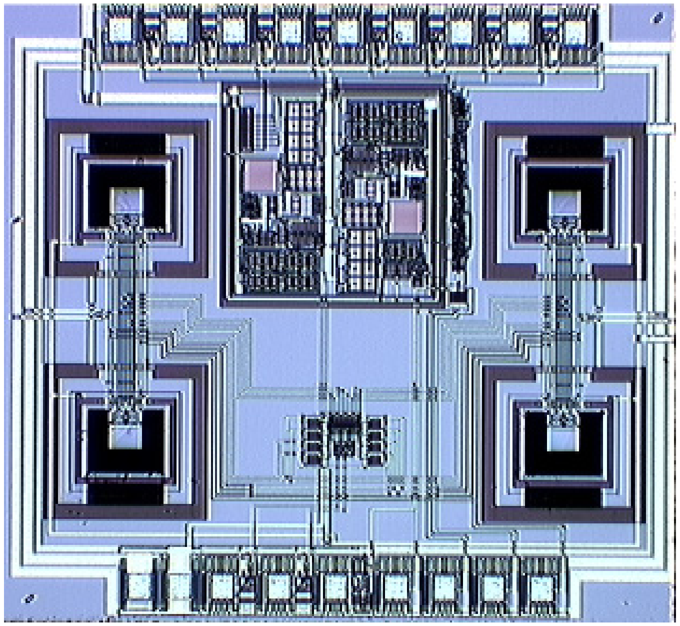
Compensated:

df/dT ≈ 0.2 ppm/˚C

O. Brand, K. Naeli et al., *Proc. MME 2008 Workshop,* pp. 121-127, 2008

Resonant BioChemical Sensors

- **Approach:** Recognition film deposited onto microresonator ad-/absorbs analyte, thus lowering the microstructure's resonance frequency
- **Implementations:** Acoustic wave devices, tuning forks, cantilevers
- **Applications:** Chemical safety, surveillance applications, environmental monitoring, medical diagnosis
- **Sensor Arrays:** Sensor selectivity through sensor arrays coated with different recognition films



D. Lange et al.,
Anal. Chem. **74** (2002) 3084-3095

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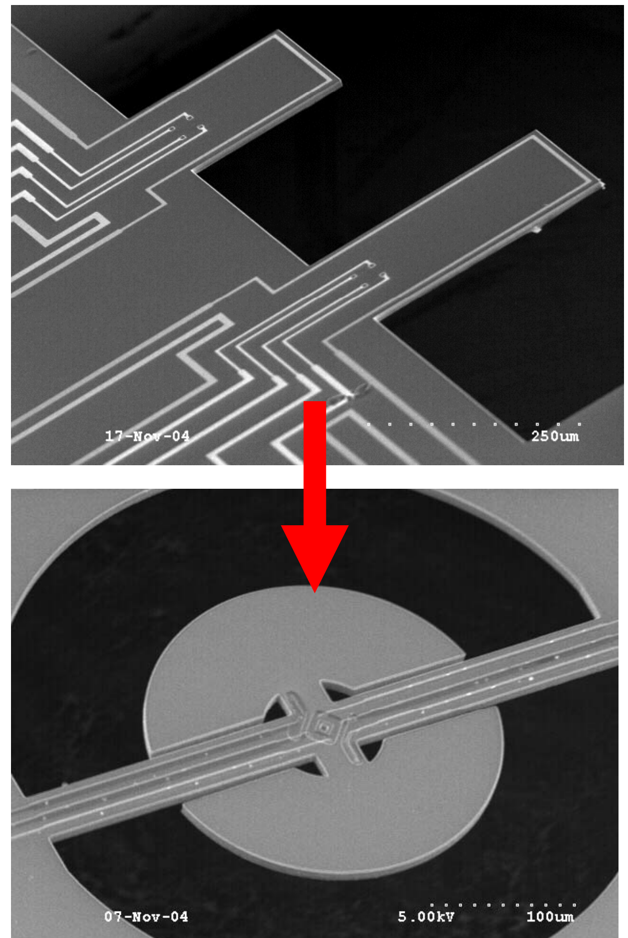
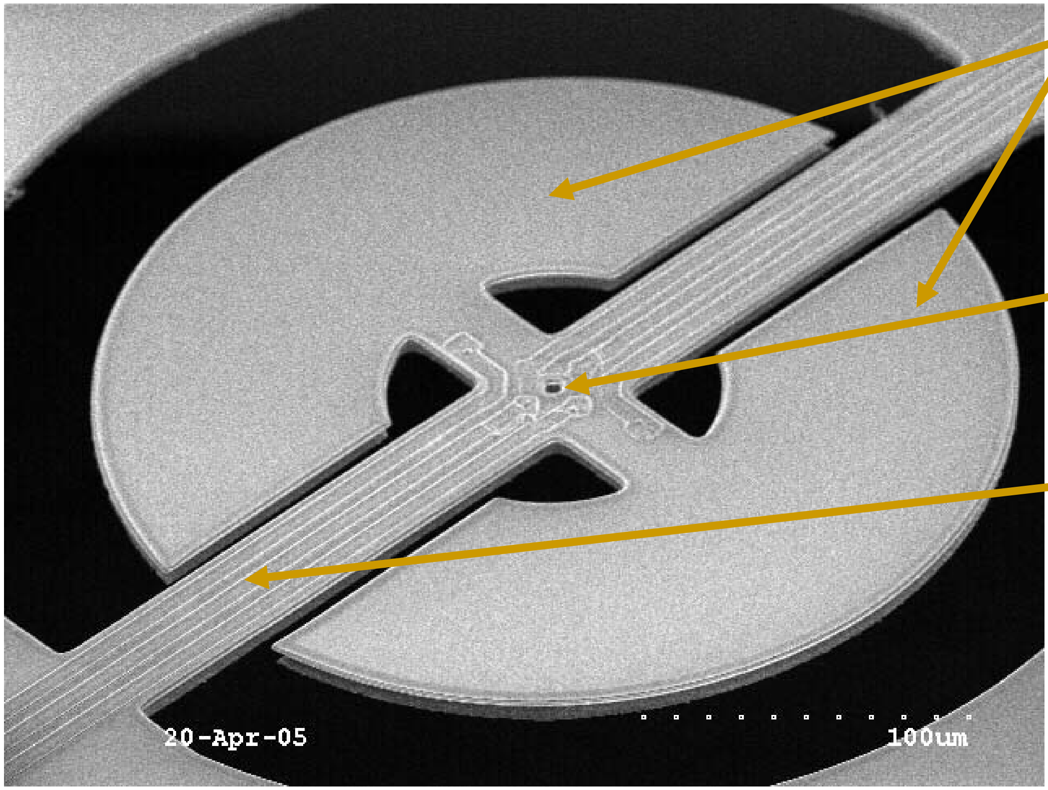
Resonant Sensor Platform for BioChemical Sensing

- **Goal:** Develop mass-sensitive sensor platform for biochemical applications **in air/liquid**
  - Large sensing area
  - High Q-factor
  - Dynamic instead of static sensing principle
- **Approach:** Investigate in-plane (instead of out-of-plane) vibration modes to improve Q-factor
- Performance achieved so far:
  Q ≈ 5800 in air at f ≈ 620 kHz



Disk-Shape Resonator



Semi-discs to be coated with sensitive layer

Center of rotation with sensing/actuation elements

Support beam

Frequency ≈ 400-600 kHz
Sensitivity ≈ 1 Hz/pg

J.H. Seo, O. Brand, *IEEE J. of MEMS* **17** (2008) 483-493

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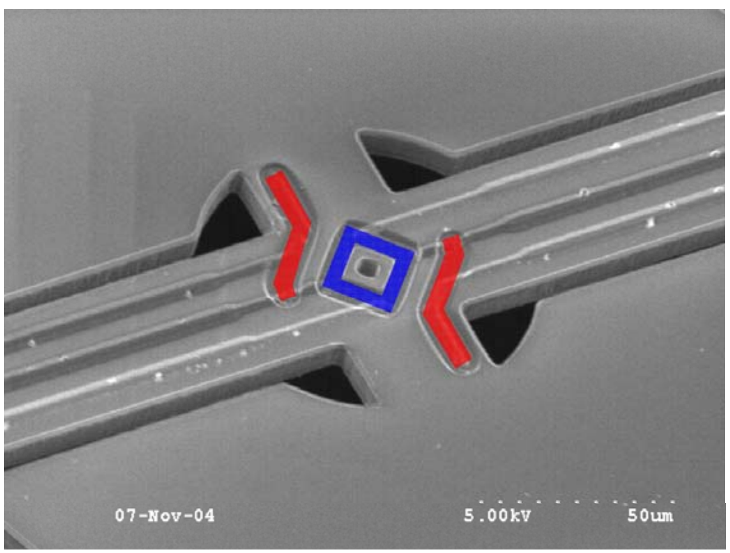
Driving and Sensing Structure

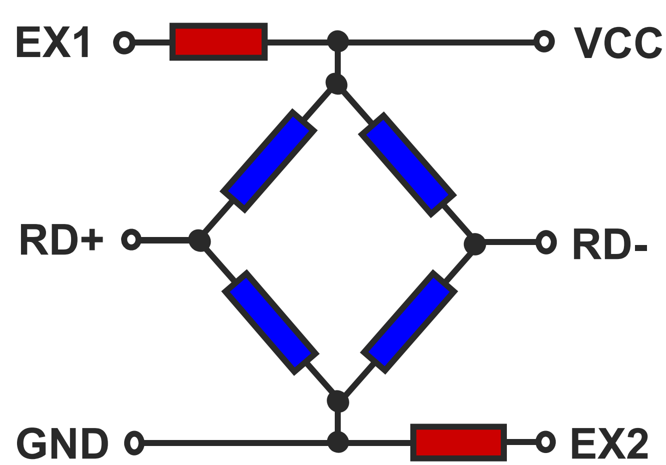
Thermal Actuation

- Asymmetric arrangement of two heating resistors
- Thermal expansion coefficient difference of Al and Si

Piezoresistive Sensing

- Wheatstone bridge sensitive only to desired in-plane vibration mode

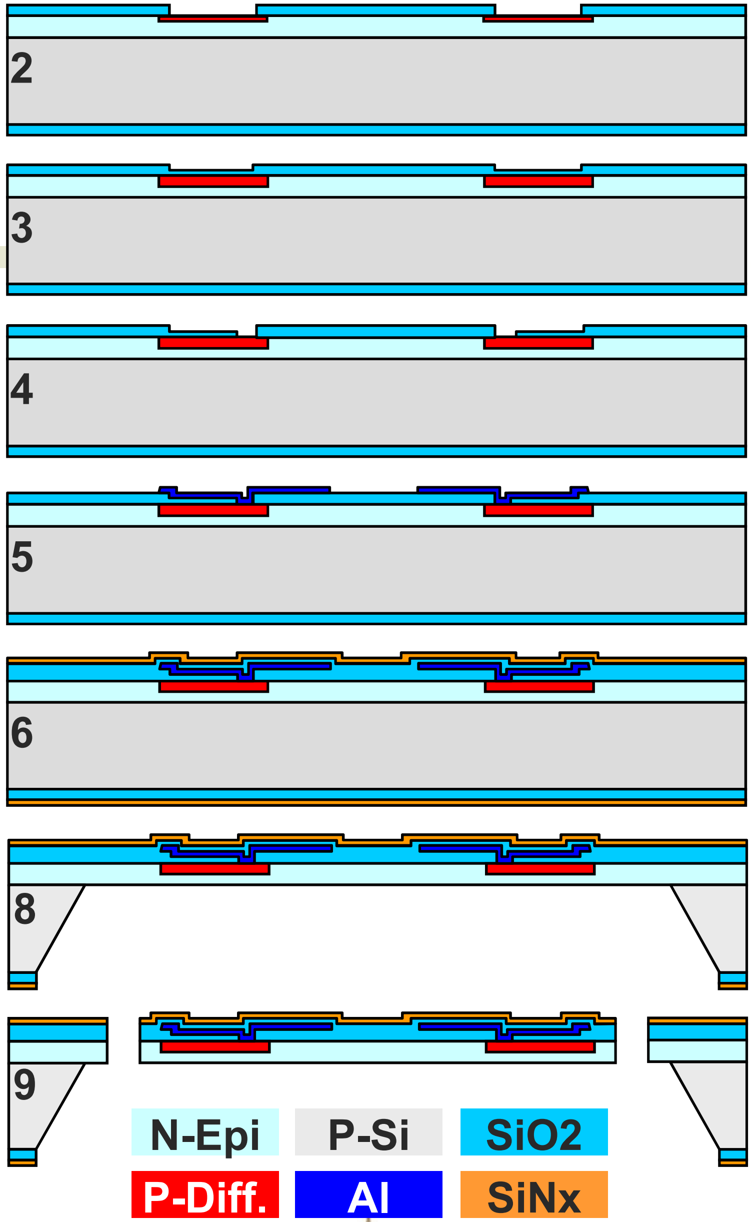




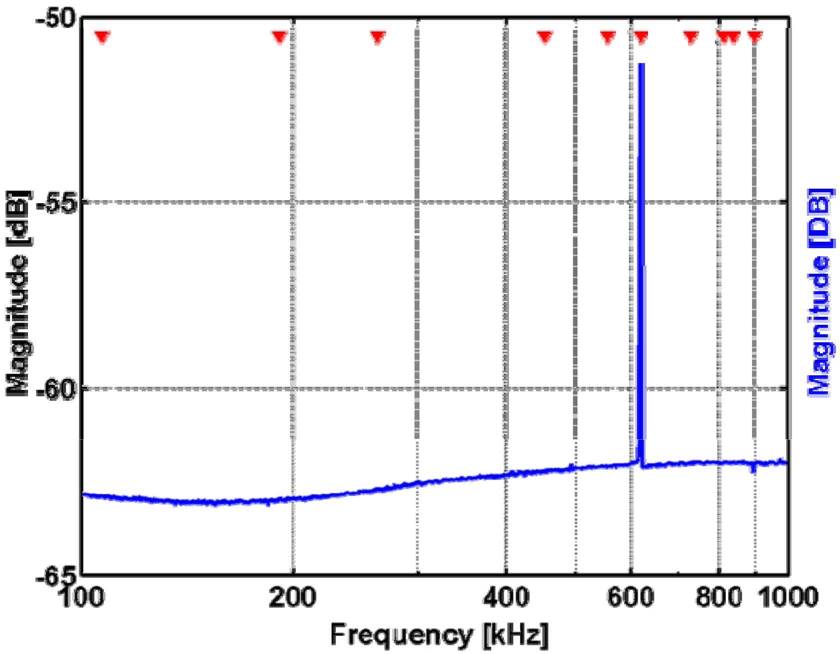
Fabrication Flow

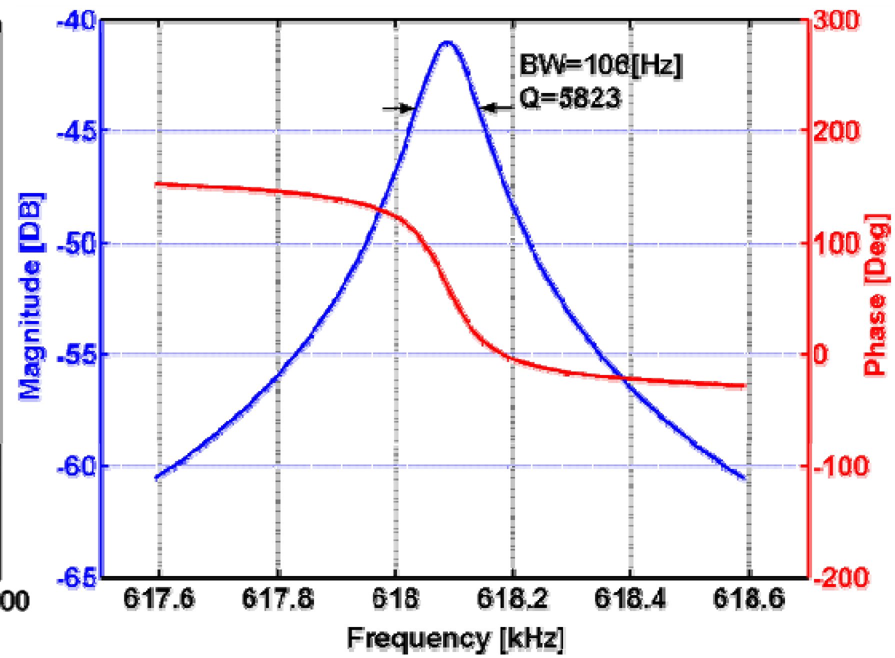
Substrate : Epi-wafer (5-10 μm n-layer)

1. Wet thermal oxidation
2. Pattern oxide & boron diffusion
3. Thermal oxidation
   (Drive-in & oxidation)
4. RIE etching of contact openings
5. DC sputter & pattern Al
6. PECVD oxide & nitride deposition
7. Pattern back side nitride & oxide layer
8. Anisotropic KOH etch
9. Release structure with RIE



Transfer Characteristic





Quality factor in air up to 5,800; in water up to 100

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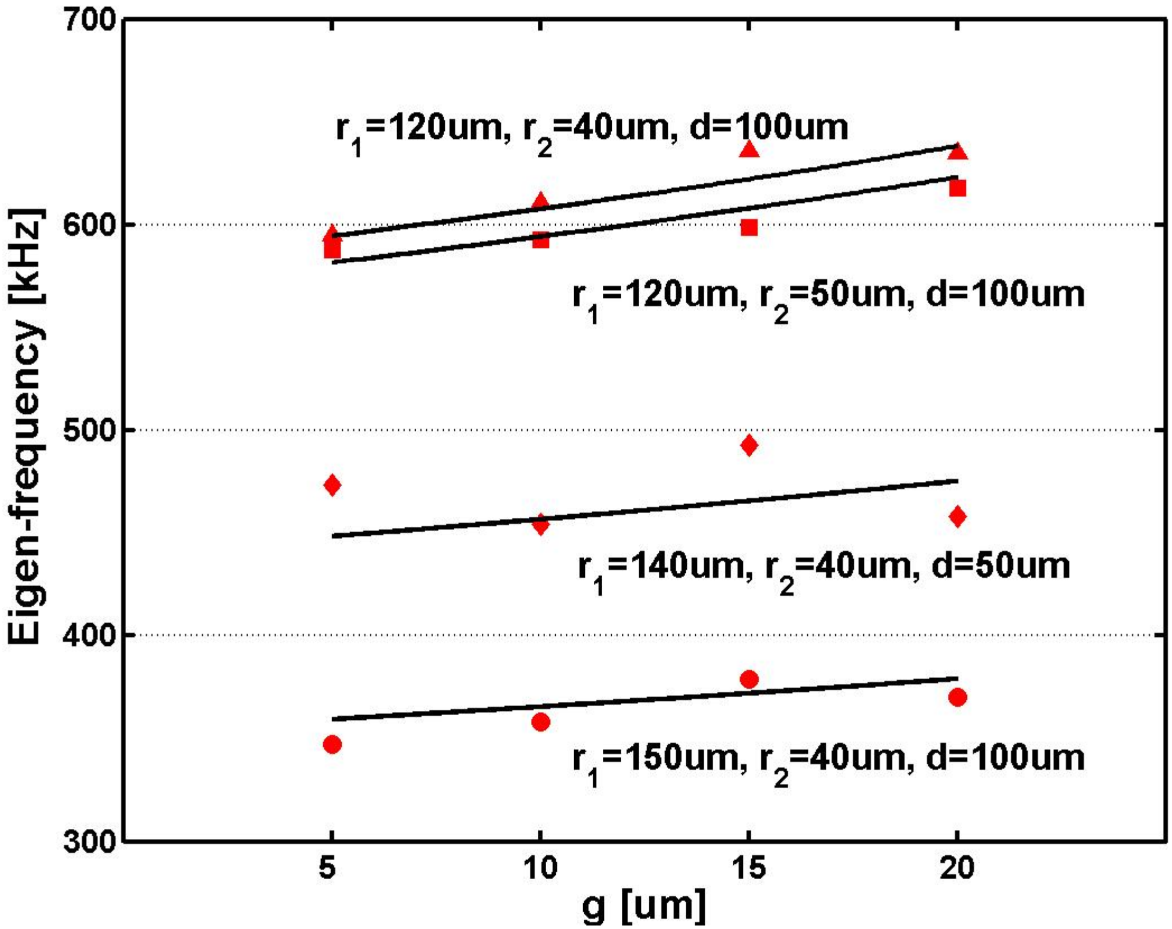
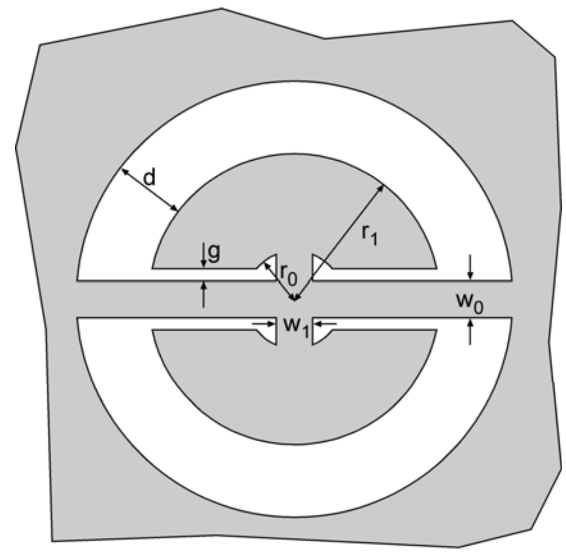
Resonance Frequency

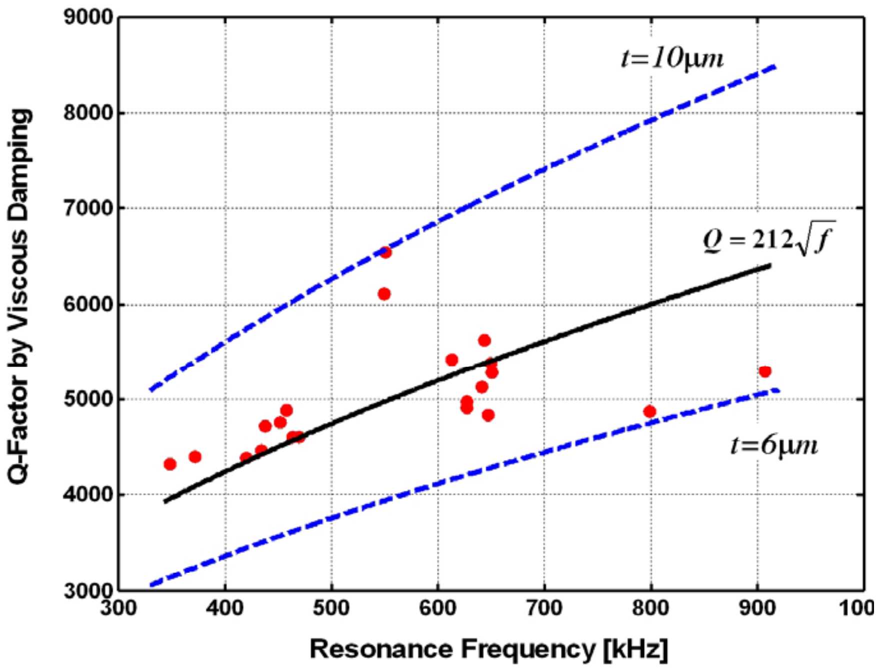




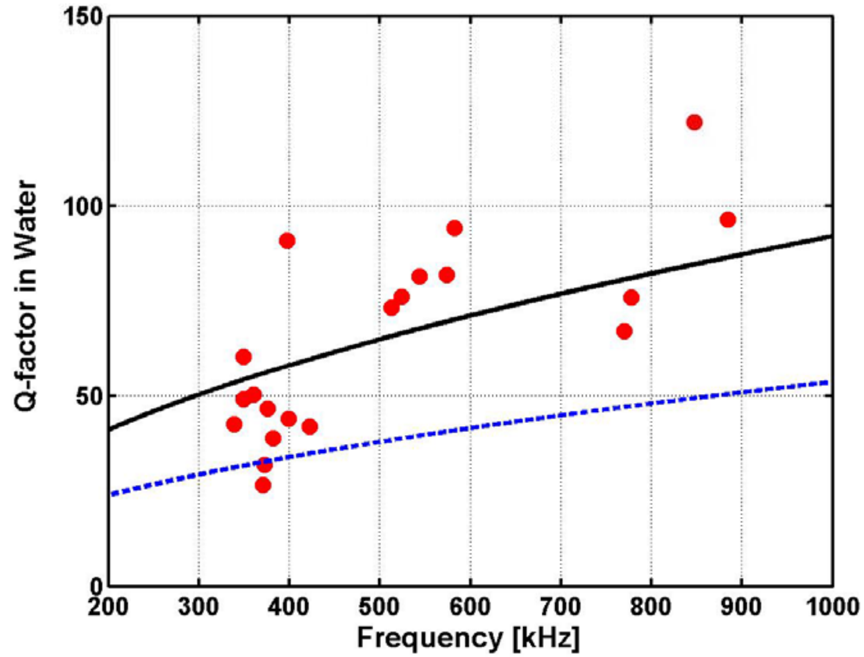
J.H. Seo, O. Brand, *IEEE J. of MEMS* **17** (2008) 483-493

Quality Factors in Air & Water

AIR



WATER



$$Q = \sqrt{\frac{\omega_0}{2\,\nu}}\,\frac{\rho}{\rho_{fluid}}\,t$$

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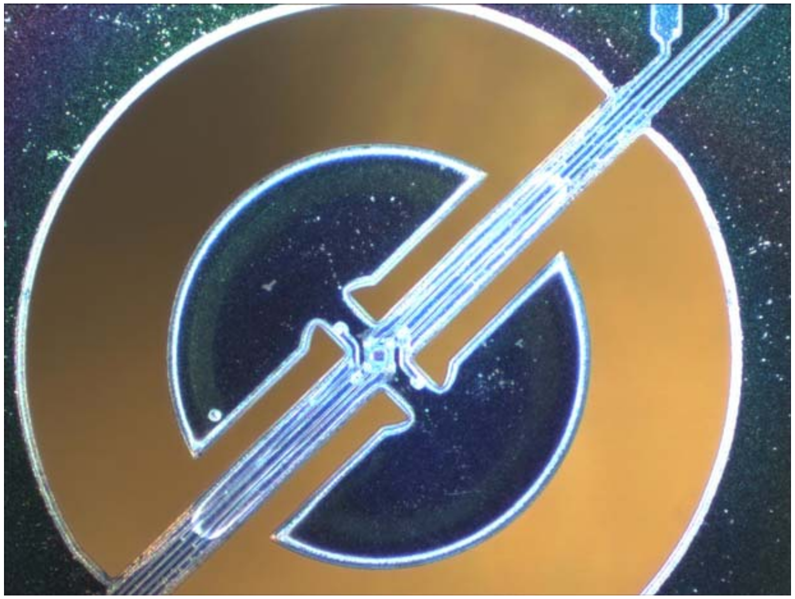
Sensitive Layer Coating

- Polymers for VOC enrichment
  - PIB: poly(isobutylene)
  - PECH: poly(epichlorohydrin)
- Polymer coating techniques
  - **Drop Coating:**
    Dispensing from syringe or BioForce Nano eNabler
  - **Spray Coating:**
    Dispensing with spray gun
- Polymer film thickness
  - 2-5 µm in air
  - < 1 µm in water



Disk resonator drop-coated with polymer film

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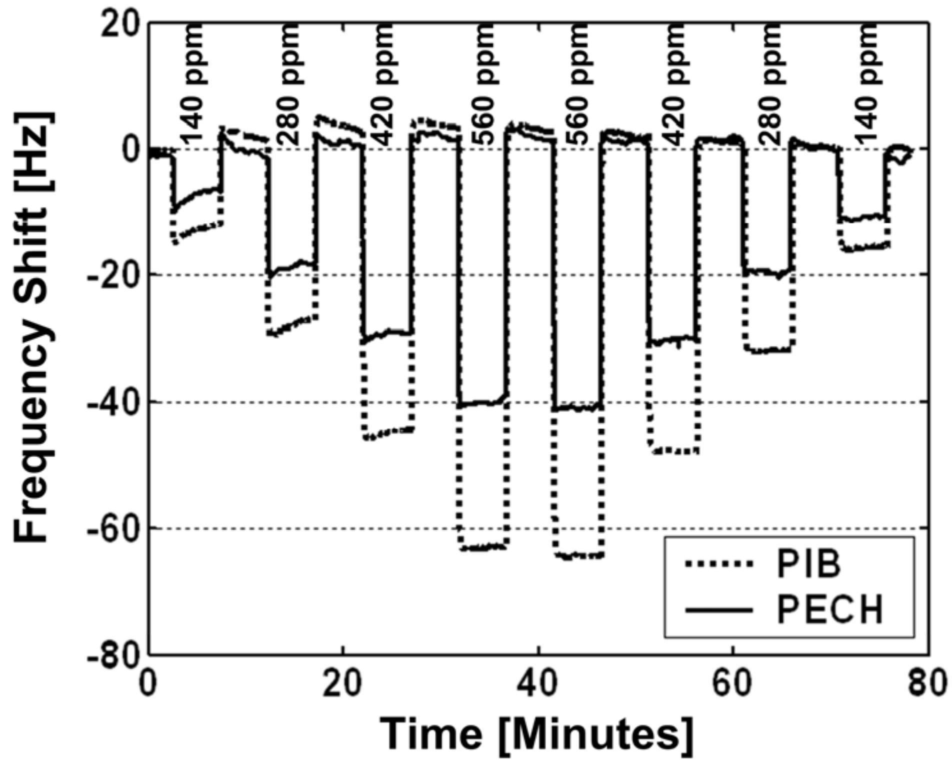
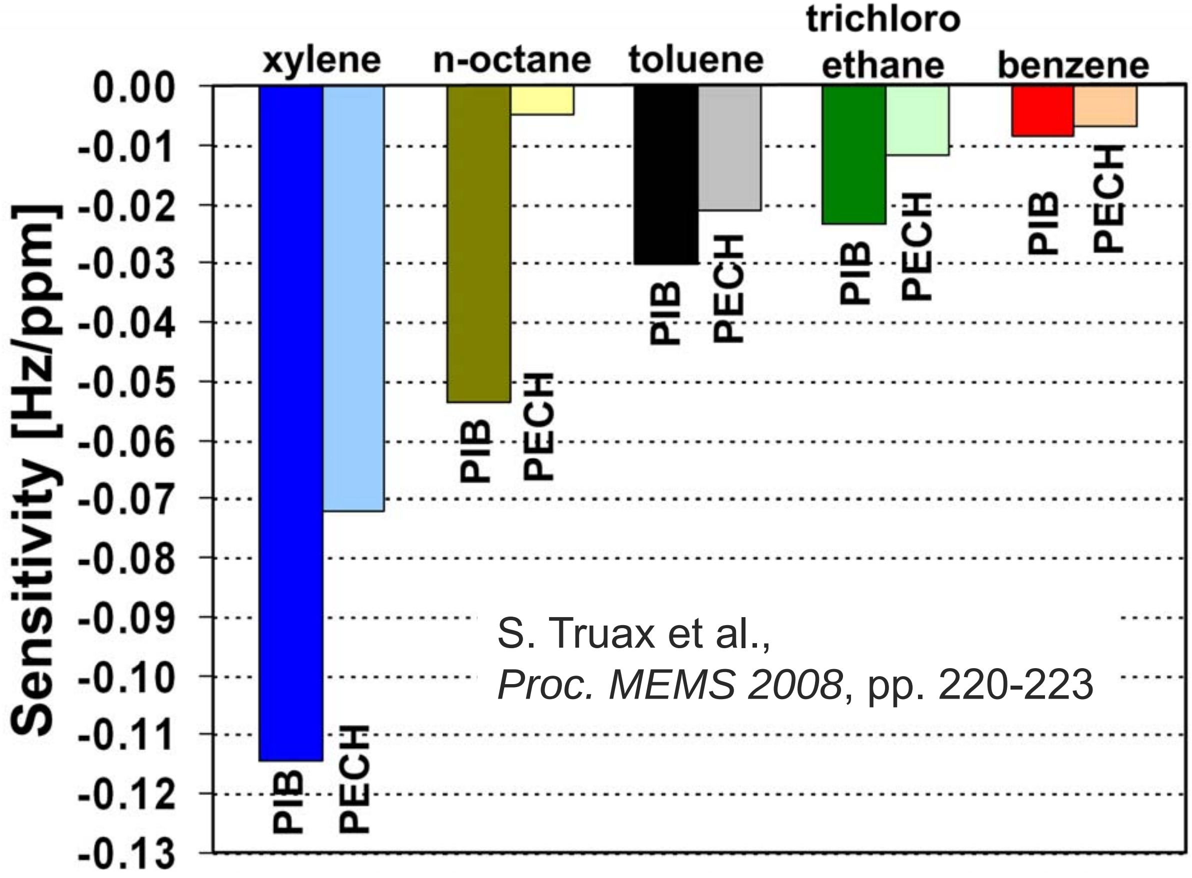
Gas-Phase VOC Sensing

- Exposure to different concentrations of o-xylene
- 4 µm spray-coated polymer films
- Turn-on and turn-off transients < 5 sec
- Short-term frequency stability ≈ 80 mHz (at 710 kHz & Q = 940)
- LOD ≈ 2 ppm for PIB/o-xylene



S. Truax et al., *Proc. MEMS 2008*, pp. 220-223

Sensitivities in Gas Phase



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Summary & Outlook

- Resonant Microsensors
  - Benefit from wide applicability and (potentially) excellent resolution
  - Frequency modulation can be introduced on “device” or “system”-level
  - Effective methods for compensating long-term drift are essential and available
- Resonant Chemical Microsystem
  - Demonstrated Q up to 5,800 in air and 100 in water
  - Demonstrated gas (and liquid-phase) chemical sensing with ppm resolution
  - Demonstrated drift compensation via controlled stiffness modulation

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- NSF for funding part of the work
  Cantilever work: 0304009
  Gas-phase Chemical Sensor: ECCS-0601467
  Liquid-phase Chemical Sensing: CBET-0606981